SUBSTITUTE SPECIFICATION WITH MARKINGS

~~WO 99/47065 AS FILED~~

~~Tooth Crowns and/or Tooth Bridges~~

BACKGROUND OF THE INVENTION

[0001] The invention relates to a process for production of an artificial tooth substitute which can be fitted on at least one preprepared dental stump, where taking into account the shrinkage, on the basis of a model, a fully ceramic skeletal structure of biologically compatible material is calculated, produced from a blank by material removal, dense-sintered and a coating material applied for individualisation. The invention also concerns a blank of porous ceramic for performance of the process.

[0002] Numerous methods are known for production of synthetic tooth crowns and/or tooth bridges. In principle after the dental preparation, a mould of the dental stump or stumps, the tooth environment and the jaw is produced. Manually by plaster moulding, a positive model of the situation of the mouth can be produced on which a skeletal structure of wax or plastic is modelled with professional skill. When conventional techniques are used, with known processes such as the lost-wax process, 1:1 copying, milling or grinding, a model of the skeletal structure can be produced in metal and over-baked with porcelain. As well as the high rejection risk in baking on the porcelain, aesthetic compromises, in particular on the cervical margin, must be made and X-ray based diagnostic processes to monitor the crowned tooth can no longer be used. So-called dental ceramic porcelains used in other processes, because of their poor mechanical properties, although suitable for tooth crowns with low loading, are not suitable for tooth bridges.

[0003] EP, B1 0389461 discloses a process for production of an artificial onlay tooth crown for fitting in a prepared tooth cavity. The process is based on a mould or negative form of the situation in the patient's mouth. Onlay tooth crowns are milled out by copy-milling, enlarged, from a green or presintered ceramic blank, and then dense-sintered. The onlay tooth crown according to EP, B1 0389461 is in principle a

different product from a tooth crown and tooth bridge as the dental indication is different. Onlay tooth crowns are fitted in tooth cavities and are always formed convex with regard to geometric shape. Tooth crowns, also with bridges, are fitted to a dental stump and have the form of a cap. This gives fine run-out margins which are technologically difficult to handle. One essential feature of copy milling according to EP, B1 0389461 is the contemporaneity of scanning and transferring the scanning movement to a machining tool. Essentially this corresponds to the working method of a pantograph which has for a long time been used for linear drawing enlargements. The area of application of EP, B1 0389461 is therefore restricted to exclusively convex formed tooth substitute products such as for example inlays, onlay tooth crowns and facings.

[0004] EP, A2 580565 describes artificial tooth restoration with powder metallurgical production processes of a ceramic, dense, high-strength skeletal structure which is coated with dental porcelain. The shape of the tooth preparation is recorded optically or mechanically in the mouth or on a tooth model. The cavities, i.e. the inner surface, and their local relation to each other are produced, enlarged, from another material e.g. plastic using a computer-controlled milling machine. The cavities of the skeletal structure are produced in a powder metallurgical process with this form, i.e. by pressing powder over a preproduced enlarged dental stump. The outer surface of the skeletal structure is also structured by pressing. The process for production of the skeletal structure thus differs in principle from production from a blank by material removal.

[0005] The inventors have faced the task of creating a process of the type specified initially which allows the production of fully ceramic tooth crowns and/or tooth bridges with a skeletal structure of dense-sintered, high strength ceramic for fitting and adhesive and/or retentive fixing on natural or artificial dental stumps. The process allows the production of tooth crowns and/or bridges with occlusal and cavital surface of materials which shrink on sintering, which have a perfect fit even with a filigree form, i.e. require no further work. Furthermore, a blank of oxide ceramic material

is provided which allows a simple precise performance of the process.

SUMMARY OF THE INVENTION

[0006] The task in relation to the process is solved according to the invention in that the three-dimensional outer and inner surface of a positive model of the skeletal structure for tooth crowns and/or tooth bridges are scanned and digitised, the data enlarged linearly in all directions by an enlargement factor f compensating precisely for the sinter shrinkage, transferred to the control electronics of at least one processing machine for machining the blank of porous ceramic and suitable tool paths derived from this, temporally decoupled from digitisation, by means of control commands for tools, material is removed from a blank until the enlarged design form of a positive model is achieved which is then dense-sintered to the skeletal structure with precise end dimensions, and then individualised by facing with a coating material of porcelain or plastic. Special and further design forms of the process according to the invention are the subject of dependent claims.

BRIEF DESCRIPTION OF THE DRAWINGS

[0007] Using the design examples shown in the drawing which are the subject of dependent patent claims, the invention is explained in more detail. Diagrammatically these show:

[0008] - Fig. 1 a longitudinal section through a natural dental stump with an artificial tooth crown,

[0009] - Fig. 2 an enlarged detail of area A according to Fig. 1,

[0010] - Fig. 3 a longitudinal section through two tooth stumps with a three-part tooth bridge,

[0011] - Fig. 4 an occlusal view of the skeletal structure of a tooth bridge,

[0012] - Fig. 5 a cavital view of the skeletal structure of a tooth bridge,

[0013] - Fig. 6 the clamping situation of a skeletal structure model for digitisation,

[0014] - Fig. 7 the clamping situation for an unmachined

blank,

[0015] - Fig. 8 the clamping situation before separation of a produced blank, and

[0016] - Fig. 9 the clamping situation for digitising a skeletal structure model of a tooth crown.

DETAILED DESCRIPTION

[0017] Starting from a dental preparation of the dental stump, a mould is made which gives a negative model of the situation in the patient's mouth, in particular the surface of the dental stump or stumps, the approximal surfaces of the neighbouring teeth and the counter-bite. Proceeding from this moulding, a positive model is produced, usually from plaster. On the positive model of the situation is applied a spacer lacquer which takes into account a gap between the surface of the skeletal structure produced on the basis of the model and the dental stump. Then on the said positive model of the situation in the patient's mouth can be produced a model for the skeletal structure of wax or plastic. This procedure is known and is used in dental technical practice for production of metal skeletal structures for tooth crowns and/or bridges.

[0018] The process according to the invention follows this known preliminary stage and digitises completely the outer and inner surface of the skeletal structure model or the surface on the positive model. A positive model reflecting incompletely the situation in the patient's mouth is preferably supplemented with regard to the three-dimensional outer and inner surface by computer technology, which is important in particular in the area of bridge elements of tooth bridges. The result of the digitisation and any computer technology supplement is a digital description of the complete surface of the skeletal structure. Digitisation can take place mechanically or optically. Processes for digitisation in the mouth of a patient on a prepared dental stump or a model are known for example from US, A 418312 (mechanical) and EP,B1 0054785 (optical). The essential disadvantage of the known mechanical digitisation lies in fixing of the mechanical scanning device to the patient, the secure handling of the

device in the narrow mouth cavity is problematical. With optical digitisation devices it is necessary to coat the dental stump with powder because of its translucent properties in order to prevent inaccuracies due to partial and uncontrolled penetration of light into the dental stump to be measured. The application of a powder coating, however, simultaneously increases the inaccuracy by application of a mostly uneven powder depth on the dental stump.

[0019] In the process according to the invention the skeletal structure model is clamped with clamping pins. The clamped skeletal structure model is suitably rotated in stages. A rotation by 180° allows a complete digitisation of the occlusally and cavitally accessible surfaces of the skeletal structure model. The optimum working positions are determined in advance and controlled by turning the shaft.

[0020] The dimensions of the surface of the skeletal structure model are enlarged linearly in all directions to compensate for shrinkage on sintering. The enlargement factor f is derived from the relative density ρ_R of the preproduced blank and the achievable relative density ρ_S after sintering according to equation 1

$$f = 3 \quad \frac{\rho_S}{\rho_R} \qquad (1)$$

[0021] From the data from the enlarged surface are generated the control commands for the machine with which the enlarged skeletal structure is produced completely and enlarged from the blank. Compared with the enlarged surface of the skeletal structure model, no machining allowance is required so that on subsequent sinter shrinkage the precise end dimensions are achieved directly, whereby retouching in the dense-sintered state is avoided.

[0022] Temporally decoupled from digitisation, a blank of porous ceramic can now be formed by material removal for the enlarged skeletal structure. For this the blank can be clamped for example between two shafts of the processing machine. The rotatably mounted blank is machined following a suitable

derived tool path. The processing can take place mechanically, for example by means of the production processes milling or grinding with one or more tools, and/or optically with one or more beams, for example by laser beams. The machining can take place in one or more processing stages, for example first rough machining and then final machining of the surfaces accessible by the tool. To change from occlusal to cavital machining, a position change of the partly machined blank may be required. The shafts holding the blank can be rotated by programmed control in steps and/or continuously, with a total of a half, a full or several rotations, including reverse rotations.

[0023] The material is preferably removed from a blank using milling tools with geometrically determined cuts at rotation speeds in the range from preferably 10,000 to 50,000 rpm, an infeed of preferably >0.5 mm, in particular 1 - 15 mm, and an advance speed of preferably >3 cm/sec, in particular 3.5 - 10 cm/sec.

[0024] The production of the skeletal structure, enlarged in relation to the positive model, from the material of the blank is completed by distal or mesial separation of the skeletal structure from the rest of the blank. At the separating points a slight manual retouching known as polishing may be required.

[0025] The finished machined enlarged skeletal structure is dense-sintered. Depending on the material used and powder morphology, the temperatures normally vary in the range from 1,100 to 1,600°C. So a density from 90 to 100% of the theoretically possible density, preferably a density from 96 to 100% of the theoretically possible density, in particular more than 99% of the theoretically achievable density can be achieved. During sintering the skeletal structure shrinks linearly without further deformation or distortion. This allows sinter baking without the sinter stump also contracting. The shrinkage S is calculated according to equation (1) from the relative density of the blank ρ_R before sintering and the achievable relative density ρ_S after sintering:

$$s = 3 \quad \frac{\rho_s}{\rho_s} \quad -1 \qquad (2)$$

[0026] After sintering, the shrunken ceramic skeletal structure is given a coating of porcelain or plastic in a conventional bake-on process at temperatures of 700 to 1100oC. One or more layers of porcelain or plastic can be applied. Thus the tooth crown or tooth bridge is given an individual appearance. The tooth crown or tooth bridge is then attached to the prepared dental stump by cement where conventional cementing materials and preparation procedures are used.

[0027] The advantages of the process according to the invention can be summarised as follows:

[0028] - High quality and precisely dimensioned, dense-sintered fully ceramic tooth crowns and/or bridges can be produced in a low cost, simple and safe process. Homogeneous blanks are essential for safe and simple production process.

[0029] - The individualised tooth crowns and/or tooth bridges to fit on prepared dental stumps resist the high loads in the side tooth area and also fulfil the aesthetic requirements of the patient in the front tooth area. In particular in the case of tooth bridges, the aim is high separation i.e. with a gracile form between the bridge elements, a structure at least comparable to metal ceramic tooth bridges can be achieved, which is required by dentists for aesthetic, hygienic and phonetic reasons.

[0030] With regard to the blank of porous ceramic, the task is solved according to the invention in that on the blank itself, its packing, an attachment label or a packing leaflet, an identification code legible by machine or with human sensory organs can be applied, which contains data for individual input of the compensating enlargement factor f.

[0031] Porous blanks of ceramic for the production of skeletal structures for tooth crowns and/or bridges can be made of various metal compositions, in particular from at least one metal oxide powder of the group consisting of

Al_3O_3, TiO_2, MgO, Y_2O_3 and ~~zircon~~ zirconia oxide mixed crystal $Zr_{1-x}Me_xO_2\text{-}(^{\underline{4n}}\ 2)_x$ where Me is a metal present in oxide form as 2-, 3- or 4-valent cation (n = 2, 3, 4 and $0 \leq x \leq 1$) and stabilises the tetragonal and/or cubic phase of the ~~zircon~~ zirconia oxide. Further details of the material composition of the blanks ~~arise from the dependent process claims 11 to 13~~ is described in detail hereinbelow. The blanks can also undergo thermal pretreatment ~~which is explained~~ as described in more detail ~~in the dependent process claims 6 to 12~~ hereinbelow.

[0032] In each of the process stages for production of a blank, tolerances apply e.g. temperature profile and temperature fluctuations during the thermal pretreatment of a blank.

[0033] The enlargement factor f (equation 1) for production of the skeletal structure from blanks is normally not a constant for the reasons given. Even if the blanks are made from one and the same material and are produced on the same production equipment with the same process, the enlargement factor f is not constant. According to the invention the flexibility in the material and the production tolerances can be achieved as the individual enlargement factor f for each blank is determined and delivered together with each blank. This is preferably achieved in that the data for the enlargement factor f is applied to be detectable optically, electromagnetically or mechanically-tactile on a blank itself, its packing, an attachment label or a packing leaflet.

[0034] According to the simplest variant, the data for the manufacture of tooth crowns and/or bridges is legible by eye and can be analysed directly or by way of an auxiliary programme for production of an enlarged design form of a positive model for a skeletal structure.

[0035] Preferably however, an identification system, known in itself, is used with which the data for the enlargement factor f can be read and automatically converted into control commands for tools.

Design Examples

[0036] A skeletal structure for a tooth bridge for

adaptation to a dental preparation is made of tetragonal stabilised ZrO_2 powder which contains 5.1 w.% Y_2O_3 and slight impurities, totalling less than 0.05 w.%, of Al_2O_3, SiO_2, Fe_2O_3 and Na_2O. The primary particle size is submicron at around 0.3 µm. The blanks are pressed isostatically at around 300 MPa and in green state the outer material layer of less than 2 mm thickness is removed by turning. After pre-processing, the diameter is 22 mm and the height 47 mm. The density is determined as 3.185 g/cm^3. The machined blank is presintered for around 120 min at approximately 850°C. After burning out the binding agents, the relative density is 3.089 g/cm^3 determined after presintering.

[0037] A mould of the situation in a patient's mouth is produced with silicon mass, in particular a negative mould of the prepared dental stump is produced with the preparation edge and approximal surfaces of the adjacent teeth. A positive form is produced by moulding in a plaster mass. To create a cement gap, the two prepared dental stumps are evenly coated on the surface with spacer lacquer and the forming surface thus structured. The skeletal structure of wax is modelled positively onto this positive form of the situation in the patient's mouth.

[0038] The wax model of the skeletal structure is clamped between two shafts and then in a serpentine manner first the occlusally accessible surface and then, after rotation of the wax model by 180°, the cavitally accessible surface is digitised mechanically. The result is a digital description of the complete surface of the skeletal structure.

[0039] The digital description is extended linearly by the enlargement factor $f = 1.2512$ calculated in formula (1) and from this are generated the control commands for the processing machine, taking into account the processing tools used for rough and final machining of the skeletal structure, which commands are then transferred to the processing machine. The end milling tools with round face have a diameter of 3 mm for rough machining and a diameter of 1.5 mm for fine machining. The partly machined blank clamped between two shafts is rotated through 180° so that the surface of the skeletal structure can be produced completely and enlarged

from the blank. Then the skeletal structure is separated from the remaining blank, the separating points on the skeletal structure is smoothed by grinding, and the skeletal structure carefully cleaned of powder residue.

[0040] The enlarged skeletal structure of ZrO_2 with Y_2O_3 is then sintered at around 1500°C. After sintering a relative density of 6.050 g/cm^3 is determined which corresponds practically to 100% of the maximum achievable density. The skeletal structure shrunken by 20.07% on sintering can be fitted to the positive model of the situation in the patient's mouth without further retouching.

~~[0041]~~ The skeletal structure is then individualised by baking on layers of porcelain at temperatures between 700 and 1100°C and affixed adhesively in the mouth of the patient with phosphate cement.

~~[0042] Using the design examples shown in the drawing which are the subject of dependent patent claims, the invention is explained in more detail. Diagrammatically these show:~~

~~[0043] Fig. 1 a longitudinal section through a natural dental stump with an artificial tooth crown,~~

~~[0044] Fig. 2 an enlarged detail of area A according to Fig. 1,~~

~~[0045] Fig. 3 a longitudinal section through two tooth stumps with a three part tooth bridge,~~

~~[0046] Fig. 4 an occlusal view of the skeletal structure of a tooth bridge,~~

~~[0047] Fig. 5 a cavital view of the skeletal structure of a tooth bridge,~~

~~[0048] Fig. 6 the clamping situation of a skeletal structure model for digitisation,~~

~~[0049] Fig. 7 the clamping situation for an unmachined blank,~~

~~[0050] Fig. 8 the clamping situation before separation of a produced blank, and~~

[0051] ~~Fig. 9 the clamping situation for digitising a skeletal structure model of a tooth crown.~~

[0052] A dental stump 10 shown in Fig. 1 has a pulpa 12 for a nerve, not shown. This dental stump is natural and vital, in other design forms the dental stump 10 can be structured

natural and non-vital or artificially on an implant. The dental stump 10 has no undercut.

[0053] On the dental stump 10 is cemented a skeletal structure 14 of dense-sintered ceramic material. This skeletal structure 14 has in the direction of an enamel 18 a fine run-out margin 16 which is essentially more difficult to produce and filigree than a known onlay tooth crown with exclusively convex surfaces. The outer surface 20 of the skeletal structure 14 runs convex and can be machined occlusally, which corresponds largely to the state of the art. The concave inner surface 22 of the skeletal structure 14 is machined cavitally which, in particular in view of the fine run-out margin 16, is extremely difficult. With the present invention this problem can be solved with the use of fully ceramic material.

[0054] To form an artificial tooth crown, coating material 24 is applied to the skeletal structure 14 until the original natural tooth form is reproduced. The skeletal structure 14 is individualised with coating material ~~20~~24, i.e. faced with porcelain or plastic.

[0055] In the enlarged area A according to Fig. 2 it is clear that further layers are formed on both sides of the skeletal structure 14 of dense-sintered ceramic. In the direction of the dentine 11 is a cement layer 26 for adhesive fixing of the skeletal structure 14 to the dental stump 10. The coating material 24 is shown only as a relatively thin layer, this layer can be essentially thicker and be structured with shaping outer surfaces 42 and thus form a tooth crown 28.

[0056] The surface 30 of the natural dental stump 10 is formed by the dental preparation. The surface 30 runs to the preparation edge 32 on which lies the fine run-out margin 16 of the skeletal structure 14.

[0057] A dental stump 10 drawn on the left in Fig. 3 largely corresponds to that in Fig. 1. A non-vital dental stump 10 drawn on the right in Fig. 3 has a lower residue of dentine 11 on which is placed an artificial dental stump 34 anchored by way of a pin 36 in the lifeless pulpa 12. On both dental stumps 10 is adhesively affixed a three-part tooth bridge 38 with a cement layer 26 (Fig. 2). This tooth bridge 38 comprises two tooth crowns 28 and a bridge element 40 which

serves as a substitute for lost tooth substance. Another three-part skeletal structure 14 of high strength dense-sintered ceramic is individualised by facing with coating material 24 of porçelain or plastic. This coating material has an outer surface 42 which corresponds as far as possible to that of the original natural teeth 10.

[0058] According to a design form not shown, a tooth bridge 38 can have more than two supporting dental stumps 10 and/or several bridge elements 40. As already stated, the supporting dental stumps 10 can also be implants with artificial dental stumps.

[0059] In the occlusal view in Fig. 4, a three-part skeletal structure 14 is shown with the outer convex surface 20, in Fig. 5 a cavital view of this skeletal structure 14 of a tooth bridge with the inner concave surface 22.

[0060] Fig. 6 shows two co-axial synchronously driven shafts 44 each narrowing at the end into a clamping pin 45. Clamped therein is a three-part skeletal structure model 46 of a tooth bridge 38 (Fig. 3) in occlusal view. The shafts 44 with the end clamping pins 45 are displaceable axially and rotatable synchronously through a preset angle. After digitisation of the occlusal side of the skeletal structure model 46, the shafts 44 are rotated through 180° and the cavital side is also digitised.

[0061] Fig. 7 shows a blank 48 of pressed ceramic powder clamped rotatably between two shafts 44. Directly on the blank 48 is applied a machine-legible information code C with data for the enlargement factor f, in the present case an electromagnetically or optically legible barcode. The information code C serves for example as identification.

[0062] For production of the blank 48, powder or colloids are processed into green blanks by way of known methods of ceramic forming. Known processes for production of ceramic green bodies are described for example in WO 94/02429 and 94/24064. For production technology reasons, geometrically simple structures such as cylinders or cubes are preferred for blanks.

[0063] Before preliminary processing, the blank 48 can be subjected to heat treatment. This is preferably carried out at

temperatures in the range from 50 to 200°C, in particular 90 to 150°C, for a duration of preferably 2 to 20 hours, in particular 2 to 6 hours. Immediately afterwards the blank 48 can be processed further with material removal to give the enlarged skeletal structure 14.

[0064] The outer layer 50 in particular is removed if the blank 48 is produced by way of pressing, casting or injection processes, in order to remove any existing density gradients in the outer material shell. Further conventional production processes for blanks 48 are cold isostatic pressing, uniaxial pressing, slip casting, diecasting, injection moulding, extruding, rolling and DCC (direct coagulation casting).

[0065] Before further processing into the enlarged skeletal structure 14, the blank 48 can undergo presintering which is preferably carried out for between 0.5 and 6 hours at a temperature of at least 450°C, in particular in a temperature range from 600 to 1200°C.

[0066] The blanks are in practice usually made of a metal oxide powder of the group consisting of Al_2O_3, TiO_2, MgO, Y_2O_3 and ~~zircon~~ zirconia oxide mixed crystal $Zr_{1-x}Me_xO_{2-(\frac{4n}{\;}\,_2)_x}$ where Me is a metal which is present in oxide form as a bi-, tri- or tetravalent cation and stabilises the tetragonal and/or cubic phase of the ~~zircon~~ zirconia oxide. In the formula for the ~~zircon~~ zirconia oxide mixed crystal n = 2, 3 or 4 and 0 < x < 1.

[0067] In a special design form, the metal oxide powder is mixed with an organic binding agent, preferably from at least one of the classes polyvinyl alcohols (PVA), polyacrylic acids (PAA), celluloses, polyethyleneglucols and/or thermoplastics. Suitably the proportion of binding agent lies in the range from 0.1 to 45 vol%, preferably in the range from 0.1 to 5 vol%.

[0068] Fig. 8 shows in occlusal view the clamping situation after processing, but before separation of the enlarged skeletal structure 14 from the remaining residue 52 of blank 48.

[0069] Fig. 9 shows in cavital view the clamping situation for digitisation of a skeletal structure model 47 for a tooth crown.

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Dear Professor Gauckler:

My name is John W. Halloran. I hold a BS Degree in Ceramic Engineering from the University of Missouri-Rolla and a PhD in Ceramics from the Massachusetts Institute of Technology. I am a Fellow of the American Ceramic Society and an Associate Editor of the Journal of the American Ceramic Society. I have been active in the field of ceramic materials for more than 30 years, during which I have published more than 100 scientific papers and patents on the subject of ceramic fabrication and sintering. My current position is Alfred Holmes White Collegiate Professor of Materials Science and Engineering at the University of Michigan, where I am the Chair of the Materials Science and Engineering Department.

At issue is if the "enlargement factor", as defined in the patent application, is specified well enough for one skilled in the art to make use of the invention. I think it is well specified. Ceramic engineers routinely consider the shrinkage during fabrication, and take this into account in designing the dimensions of the articles before sintering. Moulds, tools, CAD dimensions, etc., are routinely made larger by "enlargement factors" (or inversely "shrinkage factors"). This is a normal part of the ceramic art, and need not be specified in detail.

Also well known in the art is that the enlargement factor is computed from the starting density and the sintered density. The starting density varies in ways well known for various fabrication methods or starting ceramic powder. This is also well know for the case of ceramics produced by machining green or partially sintered blanks, and skilled ceramicists would be familiar with how to determine the relevant starting density.

EXHIBIT A

Since the starting density is an important attribute of ceramics, it is commonly measured by ceramicists. It is commonly understood that the reproducibility of the dimensions of the finished ceramic article depends upon the starting density, so efforts are made to control this factor as part of the ordinary practice of ceramic manufacture.

It is also well known that the density after sintering varies with the powder, the chemical composition, and the sintering conditions such as temperature and time, as well as other variables. The sintered density, depending upon the situation, varies from a pore-free state (theoretical density) to any chosen degree of residual porosity or relative density.

It is commonplace to choose sintering conditions to achieve the desired sintered density. The factors which influence the achievable relative density are so well-known to the ceramic engineer that it is unnecessary to specify them. Moreover, if one wishes to apply the ideas to a wide variety of ceramic materials, it is not useful to specify them in detail.

Indeed it is common for data sheets on commercial powders to list the sintered density that can be achieved under a particular sintering condition, along with other powder "specs" including purity and particle size. A person of ordinary skill in the art would not need to be taught how to determine the achievable relative density after sintering.

I am familiar with the 1996 paper by Su and Johnson on the Master Sintering Curve. This paper is a good example of attempts to supplement empirical knowledge on designing sintering heat treatments with more general treatments. As the references in this paper make clear, this effort has attracted a great deal of research for more than 50 years. It supplements but does not replace the body of empirical knowledge that is common to skilled ceramic engineers.

Skilled ceramic engineers would also recognize that the enlargement factor must be determined with better precision and accuracy if the dimensional tolerances of the sintered article are very closely controlled. Dimensional tolerances must be very closely controlled for the particular case of dental crowns.

Sincerely,

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OTHER

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To Whom It May Concern:

Letter about patent examiners office action dated June 16th, 2003

I have read the patent, and I have read the comments of the patent examiners office action dated June 16th, 2003, and I think the patent examiner is wrong. Somebody skilled in the art can readily determine the achievable relative density after sintering as it's a common knowledge and doesn't require any extra teaching. The measurement of density and shrinkage is part of the education for a ceramist. It is part of our curriculum and anyone graduating from our department can easily do the measurement and evaluation required for determining the maximum achievable sintered density. The topic is part of our ceramic processing laboratory (University of Florida, EMA 4041L) and the ceramic processing classes (University of Florida, EMA 4645, EMA 6448) that I am teaching. The students also learn how to measure the pre-sintered density and know how to calculate the required enlargement factor to achieve the required final size.

My name is Wolfgang Sigmund, I am a Ph.D. and Associate Professor of Materials Science in the Department of Materials Science and Engineering at the University of Florida. The Department is ranked among the best 9 materials departments in the nation. My qualifications in the field of materials research include ceramics, colloids, polymers, metals and processing of materials. I am a member of several leading societies, the American Ceramic Society, the American Chemical Society, the German Ceramic Society, the European Ceramic Society, the German Society for Materials, KERAMOS (ceramic honor society), and NICE (National Institute of Ceramic Engineers) and have

EXHIBIT B

been elected a member of the European Academy of Sciences. I have been an active researcher in the field of ceramic and materials research for more than 10 years, and have published more than 100 peer-reviewed research articles, in journals such as J. American Ceramic Society, Langmuir, J. Materials Research, J. European Ceramic Society, and J. of Materials Science. My work is often cited by my peers and has been recognized with over $2,100,000 in funding by DARPA, NSF, EPA and NASA as well as private foundations. I am editor for the Journal Critical Reviews in Solid State and Materials Sciences and for the special Issue Nanoparticles of the Journal for Nanoscience and Nanotechnology and am a frequent reviewer for granting agencies and journals. On the basis of my record and experience, I believe I am well qualified to comment on the patent.

Sincerely,

Wolfgang Sigmund
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April 7, 2004

To: Whom it may concern
From: Gary L. Messing, Distinguished Professor of Ceramic Science and Engineering

Subject: Expert declaration

My name is Dr. Gary L. Messing, and I am Distinguished Professor of Ceramic Science and Engineering and Head of the Department of Materials Science and Engineering at the Pennsylvania State University. I have taught Ceramic Processing to senior level students at the Pennsylvania State University since 1980 and have consulted with numerous companies involved with ceramic processing and sintering. I have published over 230 papers and co-edited 13 books on various aspects of ceramic and materials processing. I was the co-editor of the *Journal of the American Ceramic Society* from 1993-98. I am also the Editor-in-Chief of *Ceramics International* and Principal Editor of the *Materials Letters*. I was elected Fellow of the American Ceramic Society in 1990 and served on the Board of Directors from 1995-98. In 2002-2003, I served as President of the American Ceramic Society. In 2003, I was recognized as a "Highly Cited Researcher" in Materials and received the International Award of the European Ceramic Society.

I have read Patent application EP 1 067 880, "Tooth Crowns and/Tooth Bridges" and the comments of the patent examiners office action dated June 16th, 2003. The examiner writes "that the specification does not enable one skilled in the art to make or use the invention because in order to make or use the invention one must determine the enlargement factor, which cannot be readily determined because there is no teaching how to determine the achievable relative density after sintering". This statement is absolutely wrong.

The enlargement factor is used everyday at every company that manufactures ceramic components by sintering. This factor is routinely used to calculate the dimensions of the dies used to fabricate the green shaped part. Because of the variability that is intrinsic to powders, the "achievable relative density after sintering" is routinely measured to ensure reproducibility of the dimensions of the final sintering part. The measurement of "the "achievable relative density after sintering" is a standard process in industry. Companies measure density by (1) Archimedes techniques (i.e. displacement of liquid), (2) by measuring the dimensions of a simple sintered shape (e.g., a right cylinder) or (3) by dilatometry. All of these techniques are well know to those skilled in the art. These are techniques are so well established in industry, that anyone from the discipline would easily recognize the meaning of the "achievable relative density after sintering" and how to measure it.

Gary L. Messing
Distinguished Professor of Ceramic Science and Engineering

EXHIBIT C

zeichnet, dass der Binderanteil im Bereich 0.1 bis 45 vol% liegt, vorzugsweise im Bereich 0.1 bis 5 vol%.

14. Rohling (48) aus poröser Keramik zur Durchführung des Verfahrens nach einem der Absprüche 1 bis 13, **dadurch gekennzeichnet, dass** auf dem abzutragenden Rohling (48) selbst, seiner Verpackung, einer Anhängeetikette oder einem Beipackzettel ein maschinell oder mit menschlichen Sinnesorganen erfassbarer Informationscode (C) mit Daten zur individuellen Eingabe des kompensierenden Vergrösserungsfaktors (f) aufgebracht ist.

15. Rohling (48) nach Anspruch 14, **dadurch gekennzeichnet, dass** der Identifikationscode (C) optisch, elektromagnetisch oder mechanisch-taktil erfassbar aufgebracht ist.

Claims

1. Process for production of a synthetic toothless substitute (28, 38) of pressed fine ceramic powder which can fit on at least one preprepared dental stump (10), where taking into account the shrinkage the inner surface (22) of a fully ceramic skeletal structure (14) of biologically compatible material is calculated, where the geometric conditions of the patient's mouth are ~~scanned and digi~~tised, the data enlarged linear, in all directions by a enlargement factor (f), compensating precisely for the sinter shrinkage, transferred to the control electronics of at least one processing machine and suitable tool paths derived from this, the enlarged design form of the skeletal structure (14) dense sintered to the direct end dimensions and then individualised by capping with coating material (24) of porcelain or plastic,
characterised in that
on the basis of the scanning and digitisation of a positive model (46, 47) of the situation in the patient's mouth, taking into account the sinter shrinkage, an enlarged design form of the skeletal structure (14) with an inner and an outer surface (20, 22) is produced by material removal from a blank (48) of porous ceramic, where the control commands are sent to a suitable machine tool for production of the enlarged design form of the skeletal structure (14) from the blank (48) temporally decoupled from the digitisation.

2. Process according to claim 1, **characterised in that** a positive model incompletely reflecting the situation in the patient's mouth (46, 47), is supplemented by computer technology in relation to the three-dimensional outer and/or inner surface (20, 22), in particular in the area of the bridge elements (40) of tooth bridges (38).

3. Process according to claim 1 or 2, **characterised in that** the enlargement factor (f) of the positive model (46, 47) of a skeletal structure (46) is established on the basis of the material composition and powder properties, preferably according to the formula

$$f = 3\sqrt{\frac{\rho_S}{\rho_R}}$$

where ρ_R is the relative density of the preproduced blank and ρ_S the relative density achievable after sintering.

4. Process according to any of claims 1 to 3, **characterised in that** the tooth crowns (28) and/or tooth bridges (38) are formed with fine run-out margins (16).

5. Process according to any of claims 1 to 4, **characterised in that** the machined enlarged skeletal structure (14) is sintered to a density ρ_S of 90 to 100% of the theoretically possible density, preferably a density ρ_S from 96 to 100% of the theoretically possible density, in particular to a density ρ_S of over 99% of the theoretically possible density.

6. Process according to any of claims 1 to 5, **characterised in that** a green or presintered blank (48) of pressed fine ceramic powder is used, where presintering preferably takes place only after removal of the outer material layer (50).

7. Process according to any of claims 1 to 6, **characterised in that** the blank (48) is processed mechanically and/or optically, where preferably first a rough machining and then a final machining take place.

8. Process according to any of claims 1 to 7, **characterised in that** the blank (48) is subjected before pretreatment to a heat treatment at temperatures in the range from 50 to 200°C, preferably from 90 to 150°C, for a duration of 2 to 20 hours, preferably from 2 to 6 hours.

9. Process according to claim 8, **characterised in that** the blank (48) after heat treatment is processed further with material removal into the enlarged skeletal structure (14).

10. Process according to any of claims 1 to 9, **characterised in that** the blank (48), before further processing into the enlarged skeletal structure (14), undergoes presintering for 0.5 to 6 hours at a tem-

EXHIBIT D

perature of at least 450°C, preferably in the range from 600 to 1200°C.

11. Process according to any of claims 1 to 10, **characterised in that** a blank (48) of at least one of the metal oxide powders of the group consisting of Al_2O_3, TiO_2, MgO, Y_2O_3 and zircon oxide mixed crystal $Zr_{1-x}Me_xO_{2-(\frac{4-n}{2})x}$ is used, where Me is a metal which is present in the oxide form as a bi-, tri- or tetravalent cation (n = 2, 3, 4 and 0 < x < 1) and stabilises the tetragonal and/or cubic phase of the zircon oxide.

12. Process according to any of claims 1 to 11, **characterised in that** metal oxide powder with an organic bonding agent is used, preferably from at least one of the classes polyvinyl alcohols (PVA), polyacrylic acids (PAA), celluloses, polyethyleneglucols and/or thermoplastics.

13. Process according to claim 12, **characterised in that** the proportion of binding agent lies in the range from 0.1 to 45 vol%, preferably in the range 0.1 to 5 vol%.

14. Blank (48) of porous ceramic for performance of the process according to any of claims 1 to 13, **characterised in that**
on the blank (48) from which the material is to be removed itself, its packing, an attachment label or a packing leaflet, is applied an information code (C) legible by machine or with human sensory organs, with data for individual input of the compensating enlargement factor (f).

15. Blank (48) according to claim 14, **characterised in that** the applied identification code (C) is detectable optically, electromagnetically or mechanically-tactile.

Revendications

1. Procédé pour la fabrication d'une prothèse de dent artificielle (28,38) qui peut s'adapter sur un moignon dentaire (10) qui a subi une préparation préalable et qui est constituée de poudre de céramique fine comprimée, procédé dans lequel, en prenant en compte le retrait, on calcule la surface intérieure (22) d'une structure de base (14) réalisée entièrement en céramique à partir d'une matière compatible du point de vue biologique, on explore et on numérise les relations géométriques de la bouche du patient, on augmente les données de manière linéaire dans toutes les directions d'un facteur d'agrandissement (f) qui compense le retrait de frittage de manière exacte, on transfère les données dans l'électronique de commande d'au moins une machine de traitement et on en déduit des trajets d'outil appropriés, on réalise un frittage à la densité maximale de la forme de réalisation agrandie de la structure de base (14) sur la masse finale directe et on individualise ensuite ladite forme de réalisation de la structure de base (14) au moyen d'un parement avec une matière de revêtement (24) en porcelaine ou en matière synthétique,
caractérisé en ce que
sur la base de l'exploration et de la numérisation d'un modèle en positif (46, 47) de la situation dans la bouche du patient, en prenant en compte le retrait du frittage, on fabrique une forme de réalisation agrandie de la structure de base (14) qui comporte une surface extérieure (20) et une surface intérieure (22) par enlèvement de matière à partir d'une ébauche (48) en matière céramique poreuse, les ordres de commande sont envoyés à une machine-outil appropriée pour la fabrication de la forme de réalisation agrandie de la structure de base (14) à partir de l'ébauche (48) avec un décalage dans le temps par rapport à la numérisation.

2. Procédé selon la revendication 1, **caractérisé en ce que** l'on complète, par une technique de calcul, un modèle en positif (46, 47) qui reproduit de manière incomplète la situation dans la bouche du patient en ce qui concerne les surfaces extérieure (20) et/ou intérieure (22) en trois dimensions, en particulier dans la zone des éléments de pontage (40) de bridges dentaires (38).

3. Procédé selon la revendication 1, **caractérisé en ce que** le facteur d'agrandissement (f) du modèle en positif (46, 47) d'une structure de base(46) est déterminé sur la base de la constitution chimique de la matière et des caractéristiques de la poudre, de préférence selon la formule suivante :

$$f = 3\sqrt{\frac{\rho_S}{\rho_R}}$$

dans laquelle ρ_R est le poids spécifique de l'ébauche préfabriquée et ρ_S est le poids spécifique qui peut être obtenu après le frittage.

4. Procédé selon l'une quelconque des revendications 1 à 3, **caractérisé en ce que** les couronnes dentaires (28)et/ou les bridges dentaires (38) sont réalisés avec une bordure (16) qui se termine avec une faible épaisseur.

5. Procédé selon l'une quelconque des revendications 1 à 4, **caractérisé en ce que** la structure de base (14) traitée et agrandie est frittée à une densité ρ_S comprise entre 90 et 100 % de la densité théorique possible, de préférence à une densité ρ_S comprise